

Microspheres, Hollow Glass

1. SCOPE:

1.1 Form:

This specification covers hollow glass microspheres.

1.2 Application:

Primarily as a filler material in syntactic foam shapes or parts for dielectric applications.

1.3 Classification:

The hollow glass microspheres are classified according to nominal bulk density designated as 100 times the nominal weight in grams of a cubic centimeter of the microspheres.

2. APPLICABLE DOCUMENTS:

The following publications form a part of this specification to the extent specified herein. The applicable issue of referenced publications shall be the issue in effect on the date of the purchase order.

2.1 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959 or www.astm.org.

ASTM D 1214 Sieve Analysis of Glass Spheres
ASTM D 2841 Sampling Hollow Microspheres

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2.2 U.S. Government Publications:

Available from DODSSP, Subscription Services Desk, Building 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094.

2.2.1 Military Standards:

MIL-STD-794 Parts and Equipment, Procedures for Packaging and Packing of

3. TECHNICAL REQUIREMENTS:

3.1 Material:

The hollow microspheres shall be fabricated from high-purity, electrical-grade glass, forming hollow spheres 1.00 mm (0.039 inch) in diameter or smaller.

3.2 Finish:

Microspheres shall not be finished or coated. When specified, microspheres shall be finished for electrical compatibility with the resin system to be used.

3.3 Properties:

The product shall conform to the requirements of Table I; tests shall be performed on the product supplied and in accordance with specified test methods.

TABLE I

Requirement	Class Designation						Test Method
	15		22				
Bulk Density, g/cm ³	0.13	- 0.17	0.19	- 0.24			4.5.1
Particle Density, g/cm ³	0.20	- 0.25	0.30	- 0.36			4.5.2
Size Distribution, Sieve Analysis							ASTM D 1214
Weight retained on No. 80 mesh (180 μm) screen, %, maximum	6.0		6.0				
Weight passing through No. 400 mesh (38 μm) screen, %, maximum	40.0		30.0				
Surface Alkalinity, meq/g (1), maximum	0.50		0.40				4.5.3
Sinkers by volume, %, maximum	20.0		24.0				4.5.4

(1) meq = milliequivalent

3.4 Quality:

Microspheres, as received by purchaser, shall be uniform in quality and condition, smooth, and free from foreign materials and from imperfections detrimental to usage of the microspheres.

4. QUALITY ASSURANCE PROVISIONS:

4.1 Responsibility for Inspection:

The vendor of microspheres shall supply all samples for vendor's tests and shall be responsible for performing all required tests. Purchaser reserves the right to sample and to perform any confirmatory testing deemed necessary to ensure that the microspheres conform to the requirements of this specification.

4.2 Classification of Tests:

4.2.1 Acceptance Tests: Tests for particle density, size distribution, and sinker volume (3.3) and quality (3.4) are acceptance tests and shall be performed on each lot.

4.2.2 Preproduction Tests: Tests for all technical requirements are preproduction tests and shall be performed prior to or on the initial shipment of microspheres to a purchaser, when a change in ingredients and/or processing requires reapproval as in 4.4.2, and when purchaser deems confirmatory testing to be required.

4.2.2.1 For direct U.S. Military procurement, substantiating test data and, when requested, preproduction test material shall be submitted to the cognizant agency as directed by the procuring activity, contracting officer, or request for procurement.

4.3 Sampling and Testing:

Shall be as follows:

4.3.1 For Acceptance Tests: Each lot of microspheres shall be sampled in accordance with ASTM D 2841 at random throughout the lot to provide sufficient material to perform all required tests. The number of determinations for each requirement shall be as specified in the applicable test procedure or, if not specified therein, not less than three.

4.3.1.1 A lot shall be all microspheres produced in a continuous production run from the same batch of raw materials under the same fixed conditions and presented for vendor's inspection at one time. An inspection lot shall not exceed 800 pounds (363 kg) of microspheres and may be packaged in smaller quantities and delivered under the basic lot approval provided lot identification is maintained.

4.3.1.2 When a statistical sampling plan has been agreed upon by purchaser and vendor, sampling shall be in accordance with such plan in lieu of sampling as in 4.3.1 and the report of 4.6 shall state that such plan was used.

4.3.2 For Preproduction Tests: As agreed upon by purchaser and vendor.

4.4 Approval:

4.4.1 Sample microspheres shall be approved by purchaser before microspheres for production use are supplied, unless such approval be waived by purchaser. Results of tests on production microspheres shall be essentially equivalent to those on the approved sample.

4.4.2 Vendor shall use ingredients, manufacturing procedures, processes, and methods of inspection on production microspheres which are essentially the same as those used on the approved sample microspheres. If necessary to make any change in ingredients, in type of equipment for processing, or in manufacturing procedures, vendor shall submit for reapproval a statement of the proposed changes in ingredients and/or processing and, when requested, sample microspheres. Production microspheres made by the revised procedures shall not be shipped prior to receipt of reapproval.

4.5 Test Methods:

Shall be as follows:

4.5.1 Bulk Density:

4.5.1.1 Determine the weight, to the nearest 0.05 gram, of the empty graduate of a suitable volumeter, using a balance capable to weighing to 0.01 gram.

4.5.1.2 Pour a sample of microspheres, taken in accordance with ASTM D 2841, into the weighed graduate to the 100-mL mark.

4.5.1.3 Set the volumeter for 3000 taps and turn on.

4.5.1.4 Read the bulk volume of the microspheres in the graduate to the nearest 0.5 cubic centimeters.

4.5.1.5 Weigh the graduate with the microspheres to the nearest 0.05 gram.

4.5.1.6 Calculate the bulk density of the microspheres as follows:

$$\text{Bulk Density, g/cm}^3 = \frac{\text{Weight of microspheres in graduate}}{\text{Volume of microspheres in graduate}}$$

4.5.2 Particle Density: By a method agreed upon by purchaser and vendor.

4.5.3 Surface Alkalinity:

4.5.3.1 Place about 10 mL (true particle volume) of the microsphere sample in a 25-mL round-bottomed flask and add 100 mL of distilled water.

- 4.5.3.2 Boil for 1 hour \pm 0.1 under a reflux condenser.
- 4.5.3.3 Filter the suspension and wash microspheres with an additional 100 mL of distilled water.
- 4.5.3.4 Place the filtrate in a 250-mL Erlenmeyer flask and add 15 drops of methyl purple indicator solution.
- 4.5.3.5 Titrate from green to purple end point with 0.01 N hydrochloric acid.
- 4.5.3.6 Determine blank correction by repeating 4.5.3.1 through 4.5.3.5 without the microsphere sample.
- 4.5.3.7 Calculate results as follows:
- 4.5.3.7.1 Milliliters of 0.01 N HCl/100 = milliequivalents.
- 4.5.3.7.2 Milliequivalents per gram; use sample density per ASTM D 2841 to obtain true particle volume.
- 4.5.4 Sinker Volume:
- 4.5.4.1 Pour a sample of the microspheres, taken in accordance with ASTM D 2841, into a 100-mL graduate. Tap the graduate and add microspheres until the level remains constant at the 50-mL mark.
- 4.5.4.2 Add water to the 100-mL mark.
- 4.5.4.3 Shake the graduate without losing material until all glass microspheres have been completely wetted.
- 4.5.4.4 Allow the graduate to stand undisturbed for 10 minutes \pm 1.
- 4.5.4.5 Read the volume of the microspheres that have settled to the bottom of the graduate; these are the "sinks".
- 4.5.4.6 Calculate the volume of the sinks as a percentage of the original volumes of the microspheres, as follows:

$$\text{Sinker Volume, \%} = \frac{\text{Sinker volume, mL} \times 100}{50}$$

- 4.5.4.7 Report the average of three determinations.